

# Structure And Composition Of Multilaphistic Cu<sub>1</sub>/Ni<sub>1</sub>/Cu<sub>2</sub>/Ni<sub>2</sub> Chemistry Of Growth Results Using Electroplating Method In Deposition Voltage Variety As A Basic Of Low Temperature Sensor

*By* M. TOIFUR

# Structure And Composition Of Multilaphistic Cu<sub>1</sub>/Ni<sub>1</sub>/Cu<sub>2</sub>/Ni<sub>2</sub> Chemistry Of Growth Results Using Electroplating Method In Deposition Voltage Variety As A Basic Of Low Temperature Sensor

Rizalul Fiqry, Moh. Toifur, Azmi Khusnani, Yudhiakto Pramudya

**Abstract:** The deposition of Cu<sub>1</sub>/Ni<sub>1</sub>/Cu<sub>2</sub>/Ni<sub>2</sub> multilayers has been made by electroplating method in the variation of deposition voltage. This layer is a low temperature sensor material. In deposition of Ni<sub>1</sub> and Cu<sub>2</sub> deposition carried out at the electrolyte temperature of 60 waktuC in 1 minute, and electrode voltage of 1.5 volts, while the Ni<sub>2</sub> coating was carried out voltage variations ranging from 1.5 V ,5 5.5 V. The results of the study through structural tests microstructure using SEM obtained that the layer contains Cu, Ni and O. In addition, the deposition voltage of 5.5 V can increase nickel levels by up to 8%. In addition, from the microstructure test it was found that deposition stresses can shift diffraction angles, crystal structure settings and d-spacing. These three parameters have a strong influence on the multilayer chip resistivity which is minimizing Rs from 1.16×10<sup>-3</sup> Ω/sq to 1.03×10<sup>-3</sup> Ω/sq

**Index Terms:** Cu<sub>1</sub>/Ni<sub>1</sub>/Cu<sub>2</sub>/Ni<sub>2</sub> multilayers, deposition voltage, microstructure, resistivity sheet.

## 1 INTRODUCTION

The sensor is one of the detection devices that are widely used to control certain conditions. The sensors play an important role in determining the development of the world. Sensor technology is used in almost all fields of knowledge, both for research purposes and for marketing [4-7]. The stability is one parameter in sensor that determine the quality of sensor [1-3]. In Indonesia, the development of sensor technology is also relatively high. This is indicated by the rise of research on sensors, including temperature sensors. Temperature sensors are commonly used to ensure a condition is in a certain temperature range, or treat at a certain temperature. Low temperature sensors are one of a number of temperature sensors that are always constinuesly being developed [8]. The need for these low temperature sensors includes measuring the temperature of food preservatives, preserving organs, and preserving sperm of animals for breeding animals using artificial insemination methods [9]. One type of the temperature sensors that frequently used is the Resistance Temperature Detector (RTD). RTD is a temperature sensor that works based on changes in resistance due to the effect of temperature changes. RTDs are possible to be made in wire (wire wound) or thin film (thin film). In its development, semiconductor materials that have sensitivity at high temperatures are also used, for example germanium (Gr). Platinum is considered to be the best material as the basic in making RTD because of its good response, long lasting durability.

However, the problem is the price of Pt and Au that is very expensive. Another material that is possible to be used is copper (Cu) which is accompanied with nickel (Ni), either in the form of Cu-Ni alloy or Cu/Ni thin film or by making it in Cu<sub>1</sub>/Ni<sub>1</sub>/Cu<sub>2</sub>/Ni<sub>2</sub> thin film. As impurity, Ni metal has a function to increase the value of resistivity of Cu. Various studies on growing Cu/Ni thin films with electroplating methods as the base material for temperature sensors have been carried out [10-14]. In this study several variations have been carried out including time, solution concentration, voltage, and solution temperature, and annealing temperature. The ultimate goal is to determine the sensitivity of the sensor in measuring low temperatures. The commonly used medium is liquid nitrogen. But related to the physical parameters of the sensor also examined the hardness, thickness, and resistivity of the layer [15] [16]. Electroplating is a simple method for producing thin film. Electroplating is the process of deposition of anode metal ions on the surface of the cathode metal by electrolysis. During the deposition process, a chemical reaction occurs between the electrode and electrolyte [17]. In electroplating a direct current (DC) and constant voltage are needed. Coating with this method makes it possible to observe the mass transfer process of the deposit.

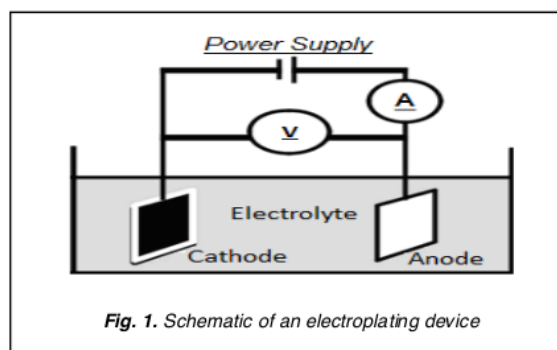


Fig. 1. Schematic of an electroplating device

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## 2 RESEARCH METHODS

### 2.1 Sample preparation

Sample preparation starts from the design of the sensor in cutting the box-shaped stickers in total size (10 x 1.3) cm<sup>2</sup>. cutting sticker is placed on a plain PCB that has a thickness of 0.2 mm Cu. PSB is then dissolved into FeCl<sub>3</sub> for 30 minutes, then cleaned with distilled water. The electroplating process was carried out in 3 stages, namely Ni coating on Cu with 60 seconds, Cu coating against Cu<sub>1</sub>/Ni<sub>1</sub> with 90 seconds to form a Cu<sub>1</sub>/Ni<sub>1</sub>/Cu<sub>2</sub> layer, and finally Cu<sub>1</sub>/Ni<sub>1</sub>/Cu<sub>2</sub>/Ni<sub>2</sub> coating. In this coating the voltage (V) varies 1.5 volts, 2.5 volts, 3.5 volts, 4.5 volts and 5.5 volts for 1 minute.

**Table 1.** Parameter for electroplating

General parameter	
Electrolyte temperature	: 60°C
Electrode distance	: 4 cm
Surface area of Ni	: 7.61 cm <sup>2</sup>
Surface area of Cu	: 7.60 cm <sup>2</sup>
Specific parameter	
Plating Ni1	: 1.5 volt, 60s
Plating Cu2	: 1.5 volt, 90 s
Plating Ni2	: 1.5 – 5.5 volt

**Tabel 2.** The concentration of the solution for plating

Plating	Material used					
	H <sub>3</sub> BO <sub>3</sub> (g)	NiSO <sub>4</sub> (g)	NiCl <sub>2</sub> (g)	H <sub>2</sub> O (ml)	CuSO <sub>4</sub> (g)	H <sub>2</sub> SO <sub>4</sub> (ml)
Ni	7,5	175	30	250	-	-
Cu	-	-	-	250	62,5	13

The test of microstructure of material was carried out using X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM), and Energy Dispersive X-Ray Spectroscopy (EDS). XRD is used to obtain data on the relationship between the intensity of diffracted rays and the diffraction angle of 2 theta [18-23]. SEM is to photograph a sample with an electron microscope that is capable in producing images with very high resolution and magnification. EDS is used to identify the chemical elements in the sample. With microscopic images produced by SEM and EDX, we can find out the topographic structure, morphology, and chemical composition of the formed layer [24] [25]. EDX aims to obtain the level of uniformity in grain size and elements found in deposits [26] [27]. Next, determine the thickness of the film by weighing the mass of Cu<sub>1</sub> as the initial mass. After coating with Ni<sub>1</sub> to Cu<sub>1</sub>/Ni<sub>1</sub> then weighed again. The calculation of the thickness of the Ni<sub>1</sub> layer is done by following the equation:

$$\delta = \frac{W}{\rho A} \quad (1)$$

where  $\delta$  is layer thickness, W the difference between Cu<sub>1</sub>/Ni<sub>1</sub> mass against Cu<sub>1</sub>,  $\rho$  Ni density, and A cross-sectional area of the layer. Likewise, to determine the variation of Ni<sub>2</sub> thickness, we weigh Cu<sub>1</sub>/Ni<sub>1</sub> / Cu<sub>2</sub> and Cu<sub>1</sub>/Ni<sub>1</sub>/Cu<sub>2</sub>/Ni<sub>2</sub> so that the mass of Ni<sub>2</sub> is the difference between the two. From the thickness of

Ni<sub>2</sub> at various electrode voltages, the effect of voltage V on Ni<sub>2</sub> thickness can be analyzed. Furthermore, the determination of the vertical resistivity (Rs) of Cu<sub>1</sub>/Ni<sub>1</sub>/Cu<sub>2</sub>/Ni<sub>2</sub> thin layer is obtained by doing a linear regression of voltage V to current I obtained from the 4 point probe. If slope of graph is a, the equation used to determine the value of resistivity is:

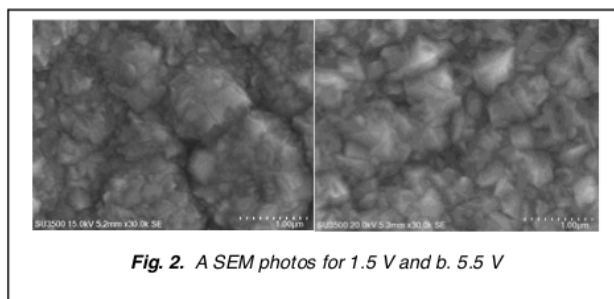
$$R_s = \frac{\pi}{\ln 2} a \quad (2)$$

By making a curve Rs vs V curve, it can be analyzed the effect of applyment V voltage on sheet resistivity of Cu<sub>1</sub>/Ni<sub>1</sub>/Cu<sub>2</sub>/Ni<sub>2</sub>.

## 3 RESULT AND DISCUSSION

### 3.1 Analysis using SEM

In figure 2, a SEM photograph<sup>5</sup>s displayed for Cu<sub>1</sub>/Ni<sub>1</sub>/Cu<sub>2</sub>/Ni<sub>2</sub> samples from Ni<sub>2</sub> deposition at a voltage of 1.5 V and 5.5 V. Based on Figure 2, there can be a morphological difference between the two samples. At a voltage of 5.5 volts, the granules appear smaller, arranged more tightly and homogeneously than the 1.5 volt voltage. it is estimated that the 5.5 volt Ni ion electrode moves faster than the 1.5 volt voltage so that the kinetic energy carried is also greater. the magnitude of the kinetic energy of an ion is able to break the bonds between the Ni deposits so that smaller granules are formed. Therefore here it appears that the plating stress affects the surface morphology of the Ni layer.



**Fig. 2.** A SEM photos for 1.5 V and b. 5.5 V

From the analysis of elemental content using EDS, it was found that the sample contained 3 elements, namely oxygen, nickel and copper. The presence of oxygen comes from H<sub>2</sub>O solvents which also react when during process of plating. But at 5.5 V deposition voltage this oxygen content decreases. Therefore the deposition voltage has an effect on reducing the oxygen content in the sample. Likewise for nickel, the levels increase with the addition of deposition voltage. This shows that Ni mass transport increases with the addition of deposition. Meanwhile at the X-r<sup>9</sup> penetrating depth the same dispersion of Ni content was accompanied by a decrease in Cu content. The increase in nickel levels is quite significant, reaching around 8%.

**Tabel 3.** he content of the chemical elements of the Cu / Ni / Cu / Ni layer from the EDX test results

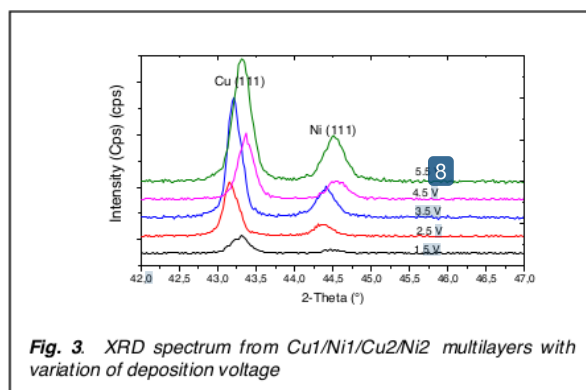
Element	% wight for 1.5 V	% weight for 5.5 V	Difference of % weight
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O K	3.7	2.69	-1.01
Ni K	86.3	94.34	8.04
sCu K	10	2.98	-7.02

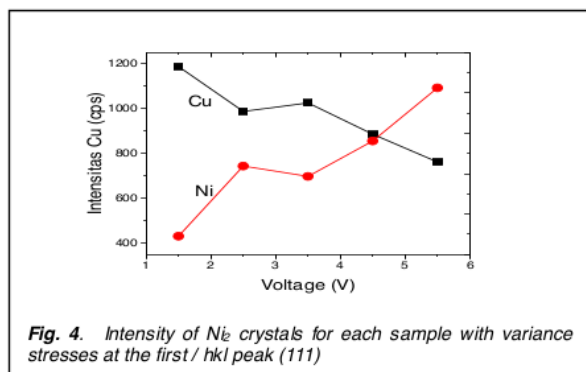
### 3.2 Analysis using XRD

results is displayed in the variation of the deposition voltage. From the picture it appears that the deposit has the form of Cu and Ni crystals with a dominant peak around around the angle of  $43^\circ$  and  $44^\circ$  in the direction (111).



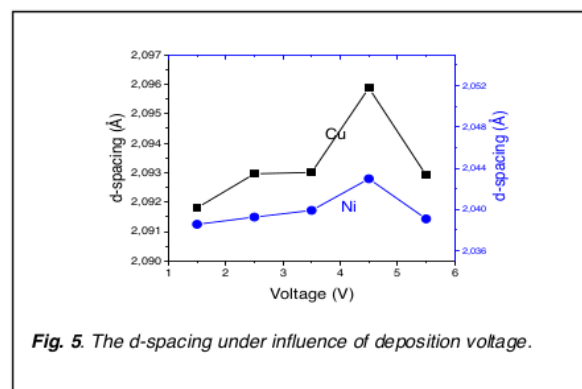
**Fig. 3.** XRD spectrum from Cu1/Ni1/Cu2/Ni2 multilayers with variation of deposition voltage

Besides that, the voltage can shift the position of the diffraction peak angle for both Cu and Ni and change the magnitude of the intensity of the diffraction peak. From the Bragg diffraction formula there is a shift in the diffraction peak related to the distance of the interplanar, whereas from the Scherrer formula it will change the grain size. To analyze in more detail the changes in the intensity of the diffraction peak, in Figure 5 an intensity curve is shown for the deposition voltage. From the picture, there is a tendency for the Ni diffraction intensity to increase, and the opposite for Cu, the diffraction intensity decreases. The decrease in the diffraction intensity of Cu is estimated to have given additional additions to the kinetic energy of Ni ions. The greater the deposition voltage the greater the kinetic power of Ni ions. When Ni ions pound the Cu surface, the Cu atoms on the surface become less organized as before.



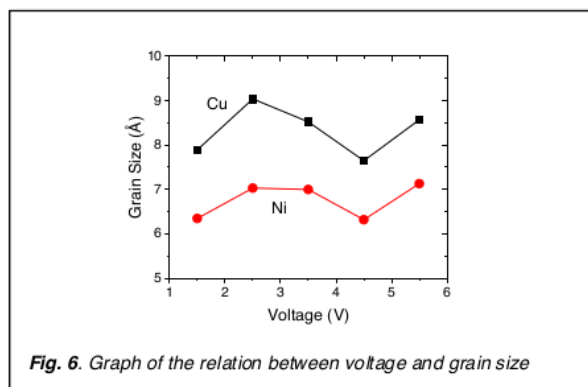
**Fig. 4.** Intensity of Ni crystals for each sample with variance stresses at the first / hkl peak (111)

In addition, the increase in deposition stress which can increase the intensity of Ni diffraction can be interpreted as accelerating the travel of Ni ions to the cathode, which further accelerates the formation of Ni deposits. This will add Ni thickness. Special conditions for the 3.5 volt voltage, Ni diffraction intensity decreased slightly from the previous 744 cps to 698 cps. It is estimated that this is related to the occurrence of hydrogen gas in the reduction reaction at the cathode so that the production of Ni deposits becomes inhibited. As a result of being inhibited, the thickness of the Ni decreases, and this can be seen at the intensity of the diffraction which is reduced.



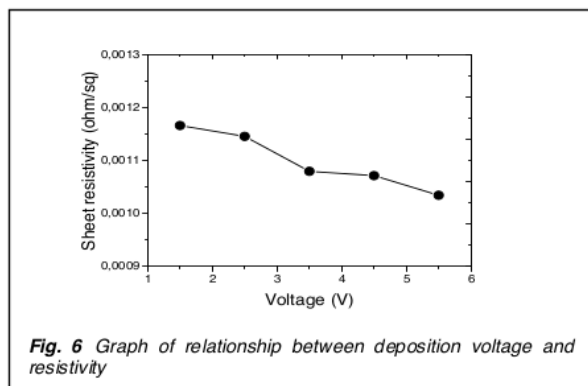
**Fig. 5.** The d-spacing under influence of deposition voltage.

In the relation to the effect of depositiona voltage on d-spacing, a curve for Cu and Ni is shown in Figure 5. From this figure, the d spacing values for Cu are higher than d-spacing Ni. This due to the atomic size of Cu (128 pm) is smaller than the size of Ni (163 pm) atoms. Besides that there is a tendency that d-spacing on the deposition voltage of 1.5 V to 4.5 V is getting greater. This is caused by hydrogen gas bubbles that form at the cathode so that in certain parts of the arrival of Ni ions at the cathode is blocked while in other parts there is no Ni hydrogen ion gas can be attached to the cathode. This will cause the formation of a layer containing porosity. In layers containing porosity, the distance between fields becomes larger. The greater the voltage the more hydrogen gas is generated so that the distance between fields is also large. If the voltage is raised again to become an overpotential, then the electroplating reaction occurs again. The 5.5V voltage is overpotential and when electroplating runs normally again the layer formed becomes increasingly continuous so that d = spacing becomes smaller again. Next, in Figure 6, the effect of deposition voltage on the grain size curve is showed. The grain size is calculated using the Scherrer formula. From the picture it appears that the deposition voltage affects the grain size. There are two things that affect grain size. Both of them compete with each other, namely the diffraction angle and the maximum half-peak width (FWHM). Shifting the diffraction angle to the left makes the grain size smaller while the effect of FWHM is the opposite, namely the greater the FWHM the smaller the grain size.



The largest grain size of 7.03 Å corresponds to a 2.5 V deposition voltage while the smallest grain size is 6.31 Å which corresponds to a voltage of 4.5 V. The increase in grain size as occurs at 2.5 V and 5.5 V is caused by the strong force of Ni ions to the Cu surface so there is a merger of Ni atoms. Besides that there is a crystal defect such as a dislocation because the subtle substrate surface makes the formation of large-sized particles to be disrupted. So small granules are formed, as happened in deposition samples at a voltage of 1.5V and 4.5V.

### 3.2 Sheet Resistivity of Cu/Ni/Cu/Ni



Microscopic parameters greatly determine the value of the sheet resistivity ( $R_s$ ). At the deposition level 1.5 volts the Ni crystal arrangement is the least regular, the d-spacing is the smallest, and the small grain size all contributes to the size of the sheet resistivity. At a 2.5 volt deposition voltage the arrangement of Ni crystals is quite regular, while the small grain size makes an increase in  $R_s$ . But small dhkl can reduce the value of  $R_s$  so that it becomes smaller than  $R_s$  at 1.5 volt deposition voltage. Thus and so on that the micro structure determines the sheet resistivity. In further research the value of  $R_s$  will become the basis for making low temperature sensors that have advantages in the microscopic parameters that determine the quality of the sensor.

## 4 CONCLUSION

From the data analysis carried out based on experimental data

and characterization tests of the structure and chemical elements of thin  $\text{Cu}_1/\text{Ni}_1/\text{Cu}_2/\text{Ni}_2$  thin layers using XRD, EDX and SEM it can be concluded that by varying the temperature sensor material obtained has superior microstructure parameters, namely crystal structure, has a smooth surface, and has a high chip resistivity.

- From the results of XRD analysis, showing the voltage that produces the best coating results is at 3.5 volts.
- The EDS results show that coating is done very well.

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